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Certified by

Jon W Dudas

Acting Under Secretary of Commerce
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Patent and Trademark Office

PROVISIONAL APPLICATION COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53 (b) (2).

Docket Number	136255-1	Type a plus sign (+) inside this box→	+

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TITLE OF THE INVENTION (280 characters max)

CUBIC BORON NITRIDE SINTERED BODY AND METHOD FOR MAKING THE SAME

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ENCLOSED APPLICATION PARTS (check all that apply)

[X] Specification	Number of pages [13]	[ ] Small Entity Statement
[X] Drawing(s)	Number of sheets [1]	[ ] Other (specify) [assignment]

METHOD OF PAYMENT FOR FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT

[ ] A check or money order is enclosed to cover the Provisional filing fees	PROVISIONAL FILING FEE AMOUNT ($)	$160.00
[X] The Assistant Commissioner is hereby authorized to charge filing fees and credit Deposit Account Number [07-0860]		

The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government

[X] No
[ ] Yes, the name of the U.S. Government agency and the Government contract number are:

Respectfully submitted,

SIGNATURE _____ DATE December 3, 2003

TYPED OR PRINTED NAME Hanh T. Pham REGISTRATION NO. (if appropriate) 40,771
PHONE NUMBER (413)-448-4664
HTP

Attorney's Docket No. 136255-1 Patent

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: Dole et al.

For: **CUBIC BORON NITRIDE SINTERED BODY AND METHOD FOR MAKING THE SAME**

Mail Stop Provisional Patent Application
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

COVER SHEET FOR FILING PROVISIONAL APPLICATION
(37) C.F.R. §1.51 (2) (i))

Sir:

Enclosed is a Provisional Application for filing. Detailed Information is set forth on page two of this cover sheet.

CERTIFICATION UNDER 37 CFR 1.10

I hereby certify that this correspondence and the documents referred to as attached therein are being deposited with the United States Postal Service on December 3, 2003, in an envelope as "EXPRESS MAIL POST OFFICE TO ADDRESSEE" service under 37 C.F.R. 1.10, Mailing Label Number EL 851486481 US addressed to Mail Stop Provisional Patent Application, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

Date: 12.3.03

Hanh T. Pham

CUBIC BORON NITRIDE SINTERED BODY AND METHOD FOR MAKING THE SAME

[001] The present invention relates to a cubic boron nitride sintered body with superior wear resistance in machining cast irons and a method of preparing the same.

BACKGROUND OF THE INVENTION

[002] Polycrystalline cubic boron nitride-based (PCBN) ceramics sintered under high pressure and high temperature are known. Sintered bodies for use in cutting tools are prepared by high pressure / high temperature sintering of cubic boron nitride (cBN) powder with a binder phase. The binder phase generally comprises ceramic materials such as TiN, TiC, TiB_2, AlN, AlB_2, or the like. The ceramic binder material, although lower in hardness and abrasion resistance than cBN, improves the performance of the tool material by reducing the amount of adhesive or chemical wear that occurs during a machining operation.

[003] Cast irons, and in particular compacted graphite irons, nodular irons, and ductile irons, can be very difficult materials to machine. PCBN tool materials are often not economical in some of these cast iron applications as rapid wear during machining leads to tool lifetimes that do not greatly exceed those of conventional materials (e.g. cemented carbides, TiC-Al_2O_3 ceramics, etc.). The use of conventional tool materials generally requires lower machining speed than what is achievable with a PCBN tool, resulting in lost productivity, lower throughput, and a higher overall part cost. It is believed that the high temperatures generated at the cutting point in some of these cast iron machining applications leads to chemical reaction between the iron and cBN grains in the PCBN. It is possible that an optimized binder composition would limit these reactions, enabling economical use of such an improved PCBN material and a resultant reduction in overall part costs.

[004] JP Patent Publication No. 07-082031 discloses a cBN sintered compact consisting of 10-70 vol. % cBN in a binding phase consisting of 5-30 vol. % of alumina (aluminum oxide) with particle diameter of 1 micron or less, 3-20 vol. % of aluminum nitride / boride; 10-40 vol. % of Ti carbide / nitride, and 3-20 vol. % of Ti boride. This sintered compact is reported to be superior to conventional products with longer service life, i.e.,

improved fracture toughness, thermal shock resistance, chipping resistance, and oxidation resistance.

[005] In another reference JP Patent Publication No. 08-126903, another different cBN sintered body with improved wear resistance is disclosed. This cBN sintered body comprises 20-40 vol. % of Ti carbide / nitride, 1-5 vol. % of aluminum nitride, 3-10 vol. % of Ti boride, 3-15 vol. % of aluminum oxide, with the residual consisting of cBN, and wherein at least 60% of the area of the cBN grains are mutually bonded.

[006] US Patent No. 5,690,706 discloses a cBN sintered compact wherein the ceramic binder consists of nanosized particles, and wherein the sintering of the compact is carried out at a relatively low pressure of less than 10 Kbar. In US Patent No. 3,852,078, a tool material is disclosed, wherein the initial sinter mixture contains hexagonal boron nitride which is converted to cBN in the sintering process. U.S. Patent No. 5,328,875 discloses a cBN sintered tool material, wherein the cBN grains are bonded with aluminum and titanium compounds including aluminum oxide, and wherein the grain sizes of all phases are less than 1 micron.

[007] There is still a need for an improved sintered compact of cBN and a binder phase that can be used to machine ferrous metals such as cast iron, compacted graphite iron, and ductile / nodular iron. Applicants have found a cBN sintered compact that exhibits excellent performance in cast iron machining applications, the cBN sintered compact comprising an optimized mixture of cBN and binder constituents wherein the cBN is homogeneously distributed within the sintered compact mixture.

SUMMARY OF THE INVENTION

[008] The invention relates to a cubic boron nitride sintered body having improved wear resistance against ferrous materials for serving as a cutting tool. The composition comprises, by volume, 10-70% cBN, homogeneously dispersed in a binder material comprising 25-50% alumina; 1-25% of at least one other aluminum compound; with the remainder comprising at least one material selected from the group consisting of carbides,

nitrides, carbonitrides, borides and silicides of group IVa, Va, and VIa of the periodic table, mixtures thereof, and unavoidable impurities.

[009] The invention further relates to a method for making a sintered compact composition comprising the steps of preparing a homogeneous powder mixture comprising, by volume, 10-70% cBN, 25-50% alumina, 1-25% of a source of metallic aluminum, with the remainder comprising at least one material selected from the group consisting of carbides, nitrides, carbonitrides, borides and silicides of group IVa, Va, and VIa of the periodic table, mixtures thereof, and inevitable impurities, and sintering the mixture into a coherent body at a pressure of at least 3 GPa and a temperature above 1000 °C.

[010] The invention further relates to the application of these sintered compacts in machining nodular irons, compacted graphite irons, grey cast irons, white cast irons, ductile irons, and spin-cast grey irons.

BRIEF DESCRIPTION OF THE DRAWING

[011] Figure 1 is a scanning electron microscope ("SEM") photograph of one embodiment of the cBN sintered compact of the present invention, showing the cBN grains to be homogenously distributed in the mixture (black grains in the structure) with very little grain to grain contact at the grain boundary.

DETAILED DESCRIPTION OF THE INVENTION

[012] The invention relates to a sintered body of cubic boron nitride (cBN) that displays surprising performance in machining ferrous materials at high cutting speeds, with an optimized composition of cBN powder in a binder comprising alumina, aluminum or aluminum compounds, and the remainder comprising carbides, nitrides, or borides of metals of groups IV-VI of the periodic table, and mixtures thereof.

[013] cBN component. cBN is indispensable in the sintered compact of the invention for imparting excellent properties of wear resistance and chipping resistance to the high pressure-sintered materials, in an amount of about 10 to up to 70 volume percent. In a

second embodiment, the cBN is present in an amount of about 15 – 40 volume %. In a third embodiment, in an amount of 15 to 25 vol. %.

[014] "cBN" as used herein refers to cubic boron nitride, wurtzitic boron nitride, or mixtures thereof.

[015] In one embodiment of the invention, the cBN has an average grain size of less than about 50 microns. In a second embodiment, the cBN has an average grain size of at least 1 micron. As finer-grained compacts give greater impact resistance and perform suitably in aggressive cutting applications, and give smoother surfaces in finishing applications, a cBN grain size less than about 30 microns is preferred. In yet another embodiment, the cBN has an average particle size of about 1 to 15 microns.

[016] In yet another embodiment of the invention, the cBN may be a mixture of uncoated cBN and coated cBN particles, or solely cBN particles coated with at least a layer of coating having a thickness from 1-50 μm. When there are multiple layers, each layer may consist of a different material. In one embodiment, the inner coating layer adjacent to the cBN particle is a material, which has a coefficient of thermal expansion closest to that of cBN.

[017] In embodiment, the coating is of multilayer type comprising combinations of any number of layers, at least three, of M(N,C,O) where M is a metal from groups IVa to VIa of the Periodic Table, and Al_2O_3. In another embodiment, the coating comprises carbide formers or coating materials which form borides or nitrides. Examples of suitable metals for coating cubic boron nitride include tin, lead, antimony, or nitrides thereof; cobalt; tungsten; titanium; zirconium; hafnium; vanadium; niobium; tantalum: chromium; molybdenum; nickel; tungsten; or a carbide, boride, nitride, or oxide thereof. In yet a third embodiment, the coating is of high adhesive strength, containing at least one layer containing at least one element selected from the group consisting of Group 4a, 5a, 6a elements, Al, B, Si and Y and at least one element selected from the Group consisting of C, N and O and having a hardness of a Knoop Hardness Hk of at least 2,000 and a film thickness of at least 0.5 μm to 10 μm.

[018] Non Aluminum Binder Materials. With respect to CBN sintered compacts, in order to produce a sintered compact with excellent properties as a cutting tool, there will be

required a binder material which provides hardness and toughness, while minimizing the tendency for chemical reaction with the workpiece under the high temperatures generated at the cutting point during a typical machining operation.

[019] Examples of non-aluminum binder materials for use in the sintered compact of the invention includes selected carbides, nitrides, carbonitrides, borides and silicides of IVa (Ti, Zr, Hf), Va (V, Nb, Ta) and VIa (Cr, Mo, W) group transition metals of the periodic table, mixtures thereof as well as solid solutions of these compounds, in an amount ranging from 5 to 40 volume %. These compounds have in common high hardness values, high welding points, and metallic properties as compared with oxides. Particularly, the heat conductivity of these compounds exhibits a value similar to metals.

[020] In one embodiment of the invention, the binder metal is selected from one of titanium nitride ("TiN"), titanium boride, titanium carbide ("TiC"), and titanium carbo-nitride ("TiCN"), with a grain size of greater than 1 micron.

[021] Alumina: Applicants have found that the use of at least 25 volume % alumina in the binder material in the cBN sintered compact of the invention surprisingly extends the life of cutting tools in machining ferrous materials. As alumina is known to be an inert material, it is believed that it reduces the tendency for chemical reaction of the tool material with the workpiece under the high temperatures generated at the cutting point during a typical machining operation. As used herein, alumina refers to bauxite (including both natural occurring bauxite and synthetically produced bauxite), calcined bauxite, hydrated alumina (e.g., boehmite, and gibbsite), Bayer process alumina, aluminum ore, gamma alumina, alpha alumina, aluminum salts, aluminum nitrates, and combinations thereof. The alumina source may contain only Al_2O_3, as well as one or more metal oxides other than Al_2O_3 (including materials of or containing complex Al_2O_3 metal oxides (e.g., $Dy_3Al_5O_{12}$, $Y_3Al_5O_{12}$, $CeAl_{11}O_{18}$, etc.).

[022] In one embodiment of the invention, the alumina has a submicron grain size, i.e., less than 1 microns. In a second embodiment, the alumina has a grain size between 1 and 100 um.

[023] Al Components. In one embodiment of the invention, the sintered material further comprises at least one aluminum containing compound other than alumina, i.e., metallic aluminum or aluminum compounds such as aluminum boride, aluminum nitride, $CoAl_3$, and $(Co, Ni)Al_3$, in an amount of up to 25 volume percent in total. In one embodiment of the invention, the amount is from 1 to 5 vol. %.

[024] The aluminum compounds can be added directly to the powder mix before sintering. Alternatively, the powder mix can contain a source of metallic aluminum, which under high pressure and high temperature sintering, will melt and react with the cBN and other binder phases to produce aluminum compounds (e.g., AlN, AlB2, TiAlN, etc.). It is preferable to add some portion of metallic aluminum to the powder mix as in metallic form, as the transient liquid aluminum that results during the sintering process can fill pores within the powder bed and provide for a more homogeneous sintered compact. In one embodiment, the amount of metallic aluminum added to the powder mix is in the range of 3 to 15 vol%. In another embodiment, the source of metallic aluminum is $NiAl_3$, added to the powder mix in the range of 5 to 20 vol%. In yet another embodiment of the invention, the sintered compact may comprise some tungsten carbide ("WC") impurity as debris from the milling operations. The WC amount may range from 1 to 15 wt. % of the total mixture.

[025] Process to manufacture cBN sintered compacts. In one embodiment to manufacture the sintered compact of the invention, at first a wet type pulverized mixing of the respective powders, e.g., binder materials and cBN, is carried out in a ball mill for a sufficient period of time to obtain a mixture with the cBN and alumina being incorporated as a homogenously distributed phase in the sintered compact. In one example, the wet blending is carried out for 30 minutes to 72 hours. In a second example, the ball milling is done in a steel mill using conventional tungsten carbide milling media, with a non-reactive liquid such as a light alcohol that acts as a milling fluid, for a period of 1-12 hours. Surfactants may be added to the milling fluid to improve the mixing and dispersion of cBN in the binder phases.

[026] In one embodiment of the invention, the mixture is blended such that the cBN grains are homogeneously dispersed in the binder phase with little cBN to cBN bonding or contact, as illustrated in Figure 1 of an SEM (scanning electron microscope) of a given colony

in the sintered compact, measured from a polished cross-section of the colony, with less than 20% of the cBN grains have grain-to-grain bonding contact. The higher the volume percentages of cBN in the PCBN composition, the more difficult it is to disperse the cBN in the binder phase well enough to minimize cBN-cBN grain contact in the sintered compact. When cBN volume percentages are above about 60%, coating of the cBN grains with binder materials, as discussed above, can be used to ensure that cBN grain contact is minimized. In the next step, the mixture is then dried and press-formed to form green compacts having dimension ranging from 1 to 15 mm thick, and 10 to 80 mm diameter. The pressed powder bodies and containment materials are then placed together in a high pressure sintering apparatus and sintered at a pressure of at least 3 GPa and a temperature of 1000 to 1600 °C for about 20 to 60 minutes, in one embodiment, between 1000 to 1400°C. The sintered compact contains cBN grains that are uniformly dispersed in the ceramic binder phase. In another embodiment, the mixture is dried and the resulting powder is loaded into a shallow, flat-bottomed, cup made of cemented tungsten carbide. The cup is covered with a refractory metal disc and sintered as above but between 1000 to 1600°C. After the sintering cycle is complete and the cup removed from the high pressure apparatus, it can be machined to form a disc of PCBN supported on a tungsten carbide substrate. In yet another embodiment, the cup is made of a refractory metal and a tungsten carbide disc forms the covering lid. This arrangement can similarly be used for fabrication of PCBN in a tungsten carbide supported form.

[027] Specimens are then machined via processes known in the art, e.g., EDM, EDG, or other processes, into a desired shape, e.g., an 80° triangle, forming a tip to use in various cutting, machining, and drilling applications.

[028] In one embodiment of an optimized sintered compact of the present invention, the material has a composition of about 15-25 vol. % cBN with an average particle size greater than 1 micron; 25-50 vol. % alumina (aluminum oxide) with submicron grain size, 8 – 40 vol. % of a non-aluminum binder material selected from carbides, nitrides, carbonitrides, borides and silicides of IVa (Ti, Zr, Hf), Va (V, Nb, Ta) and VIa (Cr, Mo, W) group transition metals of the periodic table and mixtures thereof, and 1 to 5 vol. % of metallic aluminum, aluminum compounds, or mixtures thereof.

[029] In tests employing cutting tools fabricated from the sintered compacts having the optimized composition of the present invention, the tools consistently outperform tools employing sintered compacts of the prior art in machining cast irons at high speeds in either a continuous or interrupted cutting mode.

[030] EXAMPLE. The examples below are merely representative of the work that contributes to the teaching of the present invention, and the present invention is not to be restricted by the examples that follow.

[031] In the examples that follow, some of the comparative examples employ sintered compacts that are commercially available from a number of sources, including GE Superabrasives, Inc. of Worthington, OH.

[032] Also in the examples, formulations as indicated in the tables contain cBN with an average particle size of 1-20 microns. The binder constituents have particle sizes ranging from 0.1 to 5 microns. The mixing is carried out by ball milling in a steel mill using a WC milling media with light alcohol as a milling fluid for 1-12 hours. The processed powder mixture is loaded into refractory metal cups (tantalum or niobium). The powder is leveled in the cup and a WC substrate is loaded into the cup to enclose the powder within the cup. The blank is then loaded into a high pressure cell and subject to a pressure of 40-55 Kbar and at a temperature of about 1400°C for 30-40 minutes to sinter the powder mixture and braze it to the WC substrate.

[033] Discs of 58 mm in diameter and 1.6 mm thick are formed, machined to desired geometry, e.g., 80 or 35° triangles, brazed into a corner of carbide insert bodies, and used to machine cylinder liners made out of cast iron.

[034] Flank wear is measured at a speed of 2200 surface feet / min., a feed rate of 0.10" per revolution, and 0.02" depth of cut. Each test is conducted twice and an average number is obtained.

[035] In table 1, the flank wear measurement is taken for the same amount of linear inch machined by 2 different tools, an 80° trigon and a 35° diamond. In this table, the lower the flank wear number, the better is the tool. The machining is against 10 CGI (compacted

graphite iron) cylinder liners. In some of the examples, the tool is coated with a TiC-aluminum oxide coating.

Table 1

Run #	Vol % TiC	Vol % Alumina	Vol % Al	Vol % cBN	Insert Status	Flank Wear in 0.001" 80 deg tool	35 deg tool
2	31.875	28.125	15	25	Uncoated	5.7	7.5
5	29.25	32.5	3.25	35	Uncoated	7.5	5.6
7	38.25	42.5	4.25	15	Uncoated	5.9	4.7
9	27	11.25	6.75	55	Uncoated	7.2	9.2
11	42.5	21.25	21.25	15	Uncoated	11.6	9.4
18	22.5	11.25	11.25	55	Uncoated	13.9	Broke
26	23.375	20.625	11	45	Uncoated	14.6	12.2
28	54.375	9.375	11.25	25	Uncoated	5.6	17.8
33	33.75	0	11.25	55	Coated	6.4	Broke

[036] In table 2, tool performance is expressed as linear inches machined per mil (0.001 inch) of flank wear on the tool. The greater the number of linear inches machined per 0.001" of flank wear, the better is the tool. The machining is against cast iron cylinder liners made using a spin cast process. Cast iron cylinders made via the spin cast process typically wears out cBN tools five times faster than cast iron liners made by a conventional process.

Blend #	Vol % TiN	Vol % TiC	Vol % WC	Vol % TaC	Vol % alumina	Vol % Al	Vol % cBN	cBN particle size μ	Linear inc. per 1 mil flank wear
2	0	21	0	0	12.25	1.75	65	6	3803
9	0	3.75	26.25	0	26.25	18.75	25	11	5656
10	37.5	0	0	18.75	0	18.75	25	11	4883
14	0	0	19.25	13.75	19.25	2.75	45	11	6009
19	0	1.75	12.25	0	12.25	8.75	65	2	4825
35	0	18.75	0	26.25	26.25	3.75	25	6	7229
38	0	16.25	22.75	0	22.75	3.25	35	6	4949

[037] While the invention has been described with reference to a preferred embodiment, those skilled in the art will understand that various changes may be made and equivalents may be substituted for elements thereof without departing from the scope of the

invention. All of the patents, patent applications, articles, and texts mentioned above are incorporated herein by reference.

CLAIMS

1. A wear resistant sintered compact for machining cast irons comprising 10-70 vol. % of cBN dispersed in a binder material comprising;

25-50 vol. % of aluminum oxide;

1 to 25 vol. % of at least one other aluminum compound and mixtures thereof;

with the remainder comprising at least one material selected from the group consisting of carbides, nitrides, carbonitrides, borides and silicides of group IVa, Va, and VIa of the periodic table, mixtures thereof, and inevitable impurities;

wherein the volume % is based on the total volume % of said sintered compact composition.

2. The sintered compact composition of claim 1, wherein cBN is homogeneously dispersed within said binder material.

3. The sintered compact composition of claim 1, wherein said at least one material selected from the group consisting of carbides, nitrides, carbonitrides, borides and silicides of group IVa, Va, and VIa of the periodic table is TiN, TiC, TiB2, or TiCN.

4. The sintered compact composition of claim 1, wherein said at least one other aluminum compound is aluminum nitride, aluminum boride, or mixtures thereof.

5. The sintered compact of claim 1, wherein the volume percentage of the at least one material selected from the group consisting of carbides, nitrides, carbonitrides, borides and silicides of group IVa, Va, and VIa of the periodic table is high enough to give the sintered compact an electrical conductivity sufficient for machining by electro-discharge techniques.

6. A method for manufacturing a sintered compact composition comprising the steps of;

providing a mixture comprising 10-70 vol. % cBN, 25-50 vol. % aluminum oxide, 1-25 vol % of a source of metallic aluminum, with the remainder comprising at least one material selected from the group consisting of carbides, nitrides, carbonitrides, borides and

silicides of group IVa, Va, and VIa of the periodic table, mixtures thereof, and inevitable impurities;

and sintering the mixture into a coherent body at a pressure of at least 3 GPa and a temperature above 1000 °C.

7. The method of claim 6, wherein the carbides, nitrides, carbonitrides, borides, and silicides are of Group IV metals.

8. The method of claim 6, wherein the mixture of cBN and binder materials is a homogeneous mixture.

9. The application of the sintered compact of claim 1 in machining ductile irons, nodular irons, compacted graphite irons, grey cast irons, and spin-cast grey irons.

CUBIC BORON NITRIDE SINTERED BODY AND METHOD FOR MAKING THE SAME

ABSTRACT:

A cubic boron nitride sintered body having improved strength and hardness for serving as a cutting tool, the composition comprises, by volume, 10-70% cBN dispersed in a binder material comprising 25-50% alumina, 1-25% of at least one other aluminum compound, with the remainder comprising at least one material selected from the group consisting of carbides, nitrides, carbonitrides, borides and silicides of group IVa, Va, and VIa of the periodic table, mixtures thereof, and unavoidable impurities. The cBN is homogeneously dispersed in the binder material as observed by SEM.

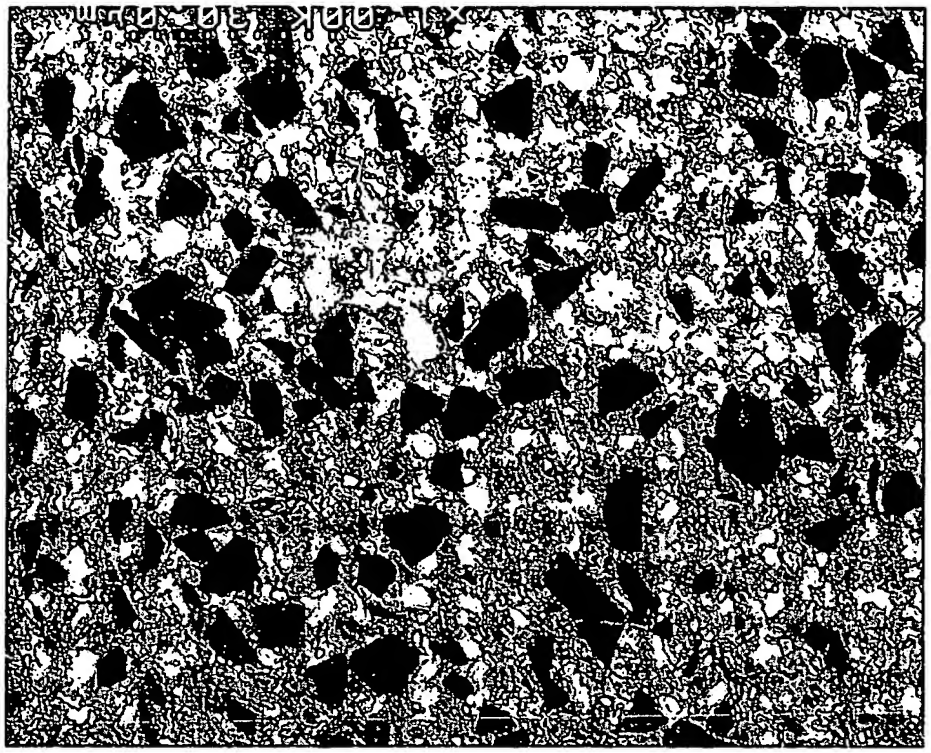

FIG. 1

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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Serial Number: N/A Filed: _____
Examiner: N/A
For: **CUBIC BORON NITRIDE SINTERED BODY AND METHOD FOR MAKING THE SAME**

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